Article

Inorganic Fertilization at High N Rate Increased Olive Yield of a Rainfed Orchard but Reduced Soil Organic Matter in Comparison to Three Organic Amendments

João I. Lopes [1], Alexandre Gonçalves [2,3,4], Cátia Brito [2], Sandra Martins [2], Luís Pinto [2,3], José Moutinho-Pereira [2], Soraia Raimundo [4], Margarida Arrobas [4], Manuel Ângelo Rodrigues [4] and Carlos M. Correia [2,*]

[1] Direção Regional de Agricultura e Pescas do Norte, 5370-347 Mirandela, Portugal; joao.lopes@drapnorte.gov.pt
[2] Centre for the Research and Technology of Agro-Environmental and Biological Sciences (CITAB), University of Trás-os-Montes and Alto Douro, Quinta dos Prados, 5000-801 Vila Real, Portugal; agoncalves@morecolab.pt (A.G.); cvqbrito@utad.pt (C.B.); scpmartins@utad.pt (S.M.); lpinto@morecolab.pt (L.P.); moutinho@utad.pt (J.M.-P.)
[3] Collaborative Laboratory Mountains of Research (MORE), Brigantia Ecopark, 5300-358 Bragança, Portugal
[4] Centro de Investigação de Montanha (CIMO), Instituto Politécnico de Bragança, 5300-358 Bragança, Portugal; sraimundo@ipb.pt (S.R.); marrobas@ipb.pt (M.A.); angelor@ipb.pt (M.Â.R.)
*   Correspondence: ccorreia@utad.pt

Abstract: Strategies for waste valorisation from domestic and agro-industrial activities must be pursued, and its use as a soil amendment is an interesting possibility. In this four-year study, the effect of applying municipal solid waste (MSW), farmyard manure (FYM), bottom wood ash supplemented with nitrogen (Ash + N), the inorganic fertilization common in the region (50 kg ha^{-1} N, P$_2$O$_5$ and K$_2$O) (Control) and this inorganic fertilization supplemented with 70 kg N ha^{-1} (High N) was assessed in a rainfed olive grove planted in a shallow soil with low organic matter and managed with conventional tillage. The High N treatment significantly increased olive yield in comparison to the other treatments (165% more than MSW), and soil available N proved to be the main driver for tree productivity. MSW and FYM increased soil organic matter, as well as the levels of phosphorus and cation exchange capacity, leaving good indications for future production cycles, although during the four years of the study these treatments provided little N to the trees. The High N treatment significantly reduced soil organic matter (63% less than MSW). The result was attributed in part to the soil management system that did not allow the development of herbaceous vegetation, but also to an effect known as "added N interaction", in which the excess of inorganic N in the soil might have contributed to accelerate the mineralization of native soil organic matter, an aspect that compromises the sustainability of this fertilization strategy. Although MSW and wood ash are sometimes associated with risks of environmental contamination with heavy metals, in this study the levels of heavy metals in soils and in plant tissues were not of concern.

Keywords: bottom wood ash; circular economy; farmyard manure; municipal soil waste; *Olea europaea*; organic manure; soil organic matter

1. Introduction

Agricultural soils contain all the essential nutrients for higher plants. However, they are not always in the most appropriate balance for plant development or in the quantities that allow high productivity to be achieved. The slush and burn system (cleaning, burning, cropping and abandonment) was the traditional way to deal with nutrient mining and allow the regeneration of soil fertility [1]. Current agricultural systems require continuous cultivation and intensification of crop production to produce more food per unit of land area. Continuous cultivation is a major cause of declining soil fertility due to the

largescale nutrient removal in crops, coupled with nutrient loss through erosion, leaching and greenhouse gas emission [2].

The fertilization strategies developed by man over time aim to restore the balance between natural inputs and outputs of nutrients, aiming at maintaining the productivity of the fields [3,4]. The intensification of agriculture has led to the generalized use of inorganic fertilizers, which are easy to apply and their nutrients readily available to plants. However, mineral fertilization is usually associated with reduced nutrient use efficiency and in some cases a high risk of environmental contamination. N fertilizers are the most controversial due to the high risk of nitrate leaching [5,6] or nitrous oxides emissions into the atmosphere [7,8]. Phosphorus can also be a delicate problem, as it is a plant-growth limiting nutrient in several parts of the world [9] and the phosphate rocks, from which P fertilizers are manufactured are a finite resource that, at the current extraction rate, is expected to be depleted within the current century [10,11]. Thus, for several reasons, it is increasingly important to reduce dependence on chemical fertilizers to restore soil fertility. On this topic, domestic and agro-industrial activities generate waste of high fertilizing value, which can contribute to reduce the dependence on inorganic fertilizers [12,13].

Farmyard manure (FYM) is the first alternative or complement to inorganic fertilizers to restore soil fertility due to its traditional abundance and ancestral use. However, in many parts of the world, as in most of the semi-arid Mediterranean regions, farms have specialized in monocultures of drought-tolerant plant species, such as vine, olive and almond, reducing livestock and consequently the availability of manure [14]. In any case, whenever available, these organic amendments must be used. From the use of manure, it is expected an increase in soil organic matter content and the enhancement of several physical, chemical, and biological soil properties [4,15].

Urban populations generate large amounts of domestic organic waste. This material can be composted, limiting their impacts in landfilling or incineration, and applied to the soil, which is in accordance with circular economy principles [16]. These fertilizing materials, usually known as municipal solid waste (MSW), may enhance soil properties and increase plant growth [15,17,18]. Still, due to the difficulty of separating organic from non-organic residues, many industrial contaminants can increase the levels in heavy metals of MSW. In the European Union, the legislation regulating the use of fertilizers may restrict or prevent the use a MSW depending on its content in heavy metals, such as cadmium, lead, chromium and nickel [19].

Fly and bottom ashes from burnt wood biomass in thermal power plants are materials of varied elemental composition, but with potential to be used in agriculture. These fertilizing materials can contain high levels of some valuable nutrients, such as calcium, phosphorus, potassium and/or magnesium [20–22], but also high levels of heavy metals [23–26] Nonetheless, several studies have shown benefits in soil properties or in the growth of agricultural and forestry plants through the application of wood ash [27–30].

In the Mediterranean basin, rainfed olive growing is usually carried out on shallow hillside soils with high risk of erosion and low levels of organic matter [31,32]. The future is challenging as growing conditions can get worse, since climate change is increasing aridity, which can reduce soil fertility [33,34]. FYM, MSW and wood ash are fertilizing resources that can be used to mitigate the degradation of soil fertility. However, even though local farmers have been using these fertilizing resources, they were not integrated into enough experimental studies, to clarify their fertilizing value and the optimal conditions for their use. Thus, in this study, the effect of FYM, MSW and wood ash on soil fertility and olive trees productive performance was compared to inorganic fertilization treatments. The working hypothesis is that these fertilizing materials can be competitive with inorganic fertilization and help to create a more sustainable cropping system within the current Mediterranean climate change scenario.

2. Materials and Methods

2.1. Study Site

The experiment was undertaken for four years (2017–2020) in a mature olive orchard with cv Cobrançosa trees, located in Mirandela (41°29′ N; 7°10′ W; 240 m above sea level), northeast of Portugal. Trees were spaced at 7 m × 7 m, corresponding to approximately 204 trees per hectare, the most common tree density in rainfed managed orchards in the region. Mirandela benefits from a typical Mediterranean climate, with an average annual air temperature of 14.3 °C and a cumulative annual precipitation of 509 mm. Average monthly temperature and precipitation for the experimental period are presented in Figure 1. The orchard is established in a bedrock of schist, loamy sand textured (6.1% clay, 17.3% silt, 76.6 sand). Some other relevant soil properties, determined from soil samples taken just before the trial started are presented in Table 1.
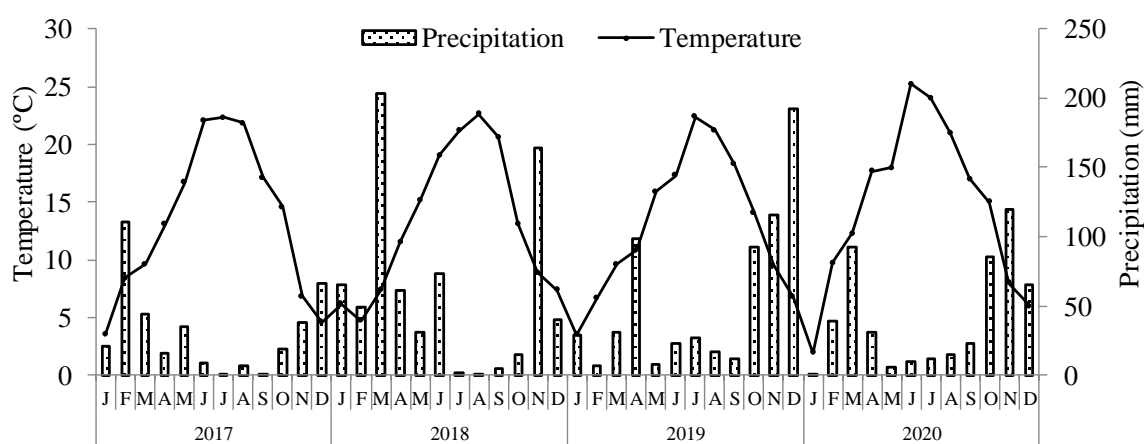

Figure 1. Average monthly temperature and precipitation during the experimental period.

Table 1. Selected Soil (0–20 cm) Properties (average ± standard deviation) Before the Trial Started.

Soil Properties		Soil Properties	
[1] Organic carbon (g kg^{-1})	8.54 ± 0.59	[5] Extract. Zn (mg kg^{-1})	1.9 ± 0.25
[2] pH (H$_2$O)	5.90 ± 0.11	[5] Extract. Cu (mg kg^{-1})	3.2 ± 0.20
[2] pH (KCl)	4.69 ± 0.09	[6] Exchang. Ca (cmol$_c$ kg^{-1})	3.88 ± 0.62
[3] Extract. P (mg P$_2$O$_5$ kg^{-1})	93.1 ± 9.5	[6] Exchang. Mg (cmol$_c$ kg^{-1})	0.71 ± 0.13
[3] Extract. K (mg K$_2$O kg^{-1})	157.6 ± 17.5	[6] Exchang. K (cmol$_c$ kg^{-1})	0.31 ± 0.05
[4] Extract. B (mg kg^{-1})	1.0 ± 0.32	[6] Exchang. Na (cmol$_c$ kg^{-1})	0.81 ± 0.11
[5] Extract. Fe (mg kg^{-1})	43.6 ± 3.25	[7] Exchang. acidity (cmol$_c$ kg^{-1})	0.08 ± 0.02
[5] Extract. Mn (mg kg^{-1})	62.4 ± 9.24	[8] CEC (cmol$_c$ kg^{-1})	5.79 ± 0.79

[1] Walkley-Black; [2] Potentiometry; [3] Ammonium lactate; [4] Hot water, azomethine-H; [5] ammonium acetate and EDTA; [6] Ammonium acetate; [7] Potassium chloride; [8] Cation exchange capacity (sum of exchangeable bases and exchangeable acidity).

2.2. Experimental Design, Fertilizing Materials and Orchard Management

The experiment was arranged as a completely randomized design with five treatments and six replications (six homogeneous trees per treatment). Between each row of marked trees of a given treatment was assigned a row of untreated trees. The treatments were: (i) the inorganic fertilization program followed in the orchard in the previous years (Control); (ii) local farmyard manure (FYM); (iii) municipal soil waste (MSW); (iv) bottom ash + inorganic N (Ash + N); and (v) the inorganic fertilization program reported supplemented with N (High N).

The control treatment was set as the inorganic fertilization program followed in the orchard in the previous years, consisting of a compound NPK fertilizer (10:10:10) applied annually at a rate corresponding to 50 kg ha^{-1} of N, P$_2$O$_5$, and K$_2$O, supplemented with 2 kg B ha^{-1} as borax. FYM was a compost resulting from sheep excreta and urine

mixed with rye straw, from a flock of sheep which graze freely during the day and spend the night in a barn. MSW is a commercial compost, Ferti-Trás-os-Montes®(Resíduos do Nordeste, Mirandela, Portugal), produced from the organic fraction of undifferentiated MSW by the intermunicipal company 'Resíduos do Nordeste', which manages waste from 13 municipalities in the northern region of Portugal. Bottom ash was obtained from a wood biomass burning plant (Biomass Thermoelectric Power Plant Terras de Santa Maria, Oliveira de Azeméis, Portugal). Properties and elemental composition of these three amendments are shown in Table 2. FYM and MSW were applied every year at variable rates, depending on dry mater yield and N concentration, in order to apply 50 kg N ha^{-1} yr^{-1}, the same rate of N of the control treatment. Wood ash was applied at a rate of 4 t ha^{-1} (dry weight) in 2017 and 2018. Although the levels of heavy metals seem safe, according to National legislative framework (Decree-Law No 103/2015 of 15 June 2015, which established the rules for placing fertilising materials on the market), it was decided to apply the bottom ash only in the first two of the four years of the study. The treatment of bottom ash was complemented with 50 kg N ha^{-1}, the N rate used in the control treatment, due to very low N content in ash. Thus, in 2019 and 2020 the plot of bottom ash received only N (50 kg N ha^{-1}, as ammonium nitrate, 20.5% N). The inorganic fertilizer applied at increased N rate (High N) consisted in the application of 50 kg ha^{-1} of N, P$_2$O$_5$, and K$_2$O as a compound NPK (10:10:10) fertilizer, supplemented with 70 kg N ha^{-1} as ammonium nitrate (20.5% N). This treatment represents a trend that exists among some farmers in the region for the intensification of the cropping system. Amendments and fertilizers were homogenously spread beneath the tree canopy, followed by incorporation into the soil with cultivator, as common in the region.

Table 2. Properties (average ± standard deviation) of soil amendments used in the field experiment.

Properties	Municipal Solid Waste				Farmyard Manure				Bottom Ash *	
	2017	2018	2019	2020	2017	2018	2019	2020	Properties	2017/2018
Dry matter (%)	68.3 ± 3.0	78.5 ± 6.5	87.4 ± 7.1	78.3 ± 6.3	34.5 ± 3.8	51.5 ± 7.5	51.1 ± 6.5	63.0 ± 3.4	Dry matter (%)	59
[1] Cond (mS cm^{-1})	5.6 ± 0.2	6.2 ± 0.3	4.6 ± 0.4	4.8 ± 0.3	5.6 ± 0.3	8.0 ± 0.7	7.0 ± 0.8	4.3 ± 0.4	Organic matter (%)	11
[2] pH (H$_2$O)	8.2 ± 0.1	7.8 ± 0.1	7.9 ± 0.1	8.1 ± 0.1	8.6 ± 0.1	9.0 ± 0.1	8.2 ± 0.2	8.6 ± 0.1	pH (23.4 °C)	12
[3] C (g kg^{-1})	236.1 ± 17.0	247.0 ± 23.1	218.1 ± 22.3	222.4 ± 15.9	306.4 ± 19.8	258.6 ± 19.3	223.6 ± 12.8	249.3 ± 55.2	Total N (g kg^{-1})	<5.6
[4] N (g kg^{-1})	17.5 ± 2.0	15.7 ± 1.3	16.8 ± 1.2	17.5 ± 1.6	22.6 ± 1.9	14.9 ± 1.2	13.7 ± 1.2	15.2 ± 1.4	NO$_3^-$-N (mg kg^{-1})	<4.5
[5] P (g kg^{-1})	4.5 ± 0.1	4.7 ± 0.2	3.2 ± 0.1	3.4 ± 0.2	6.5 ± 1.2	6.1 ± 0.4	7.4 ± 0.6	4.4 ± 0.4	NH$_4^+$-N (mg kg^{-1})	<4.2
[6] K (g kg^{-1})	14.1 ± 2.4	15.9 ± 1.6	13.6 ± 1.4	13.1 ± 2.1	55.1 ± 4.4	28.5 ± 3.8	23.5 ± 3.1	23.1 ± 2.7	P (g kg^{-1})	1.2
[6] Ca (g kg^{-1})	74.3 ± 3.0	63.0 ± 5.1	64.8 ± 4.4	27.8 ± 2.5	33.5 ± 3.3	22.3 ± 2.1	12.3 ± 1.5	18.8 ± 1.4	K (g kg^{-1})	9.7
[6] Mg (g kg^{-1})	8.3 ± 0.3	8.3 ± 0.7	7.9 ± 0.7	8.8 ± 0.8	8.5 ± 0.9	8.4 ± 1.3	9.4 ± 1.6	8.5 ± 0.7	Ca (g kg^{-1})	20
[5] B (mg kg^{-1})	49.0 ± 4.4	74.4 ± 3.7	65.3 ± 6.2	58.1 ± 4.3	46.1 ± 6.4	39.7 ± 7.1	30.7 ± 8.1	56.1 ± 7.6	Mg (g kg^{-1})	5.2
[6] Cu (mg kg^{-1})	265.7 ± 68.1	184.0 ± 33.2	169.5 ± 28.6	249.4 ± 31.8	32.8 ± 2.6	56.1 ± 4.9	36.1 ± 2.9	49.4 ± 5.1	Na (g kg^{-1})	2.1
[6] Fe (g kg^{-1})	12.3 ± 2.6	11.9 ± 2.1	12.8 ± 1.9	13.8 ± 1.2	6.1 ± 0.9	17.3 ± 1.5	17.7 ± 1.3	13.4 ± 1.1	Zn (mg kg^{-1})	47
[6] Zn (mg kg^{-1})	487.9 ± 17.0	419.0 ± 38.0	528.0 ± 41.2	428.4 ± 41.9	200.2 ± 9.5	144.1 ± 12.6	124.9 ± 11.2	228.4 ± 25.8	Cu (mg kg^{-1})	<17
[6] Mn (mg kg^{-1})	474.8 ± 31.1	569.7 ± 43.2	414.2 ± 38.5	429.1 ± 31.3	366.3 ± 37.3	479.5 ± 52.2	579.8 ± 61.1	319.3 ± 41.6	Ni (mg kg^{-1})	<10
[6] Cd (mg kg^{-1})	6.4 ± 0.8	7.2 ± 0.7	5.5 ± 0.6	5.8 ± 0.4	0.7 ± 0.0	1.3 ± 0.1	1.3 ± 0.1	1.6 ± 0.2	Pb (mg kg^{-1})	18
[6] Cr (mg kg^{-1})	57.5 ± 4.2	40.2 ± 4.1	93.8 ± 8.6	62.4 ± 6.5	35.8 ± 0.5	41.8 ± 6.9	31.8 ± 4.9	29.6 ± 3.4	Cr (mg kg^{-1})	24
[6] Ni (mg kg^{-1})	44.8 ± 2.8	77.9 ± 3.7	60.0 ± 4.1	53.2 ± 5.2	18.4 ± 1.6	18.2 ± 3.8	14.2 ± 4.2	21.8 ± 3.6	Cd (mg kg^{-1})	<0.33
[6] Pb (mg kg^{-1})	198.6 ± 60.8	149.2 ± 25.5	101.8 ± 12.5	132.6 ± 14.1	36.0 ± 3.2	33.8 ± 4.9	23.8 ± 4.7	28.6 ± 5.1	Hg (mg kg^{-1})	<0.33

[1] Conductivimeter; [2] potentiometry; [3] Incineration; [4] Kjeldahl; [5] Colorimetry; [6] Atomic absorption spectrophotometry. * Provided by the manufacturer (in 2017 and 2018 it was applied the same product).

The orchard floor was managed by conventional tillage, performed with a cultivator twice a year, between March and May, after the application of fertilizers and amendments. No relevant phytosanitary problems were detected during the experimental period, so there was no need to apply pesticides. Pruning was performed once a year, in the resting period of winter, usually in December shortly after harvest. A light pruning regime was implemented, trying to remove no more than 15 to 20% of the leaves. Pruning wood of each individual tree was weighed fresh in the field. Subsamples of ~1 kg representing all

parts of the prunings (thick and thin wood and leaves) were sent to the laboratory, weighed fresh again, oven-dried at 70 °C to a constant weight and weighed dry to allow estimating the total dry matter removed in prunings. The harvest was performed every year by late November, using a branch shaker harvesting machine to pull the fruit down, with sheets spread on the floor to recover it.

2.3. Leaf Gas Exchange Determinations

Leaf gas exchange measurements were performed during the four years of the experiment in healthy and full expanded mature leaves on cloudless mornings (photosynthetic photon flux density above 1500 μmol m^{-2} s^{-1}) using a portable IRGA (LCpro+, ADC, Hoddesdon, UK), operating in the open mode. Net photosynthetic rate (A, μmol CO$_2$ m^{-2} s^{-1}) and stomatal conductance (gs, mmol H$_2$O m^{-2} s^{-1}) were estimated using the equations developed by von Caemmerer and Farquhar [35]. Intrinsic water use efficiency was calculated as the ratio of A/gs (μmol mol^{-1}).

2.4. Samples Collection and Laboratory Analysis

Twice a year, in late July, at endocarp sclerification, and in the winter resting period of olives, leaf samples were taken from the middle part of the current season shoots in the four quadrants at approximately 1.8 m high. Leaf samples were used for elemental analysis, allowing for the monitoring of the nutritional status of trees. Pruning wood was separated into stems and leaves and weighed in the field. Subsamples of both plant parts were weighed again, carried out to the laboratory, oven-dried at 70 °C and weighed dry. In November the fruits were harvested and weighed separately per tree. The harvesting method was already described. A random sample of 30 fruits was separated for elemental analysis. All plant tissues were oven-dried at 70 °C and ground before analysis. In June 2020, the soil was sampled at three depths (0.0–0.1 m, 0.1–0.2 m, and 0.2–0.3 m) for assessing the effect of the fertilizer treatments on soil properties. Three replicates per soil layer were prepared after taking soil from 10 different points (composite samples).

In the lab, soil samples were oven-dried at 40 °C and submitted to the following analytical determinations: (1) pH (H$_2$O and KCl) (potentiometry); (2) organic C (Walkley-Black method); (3) exchangeable bases, acidity and cation exchange capacity (ammonium acetate, pH 7.0); (4) extractable P and K (ammonium lactate solution at pH 3.7); (5) extractable boron (B) (hot water, and azomethine-H method); (6) extractable Fe, Mn, Zn, Cu, Ni, Cd, Cr, and Pb (ammonium acetate and EDTA, determined by atomic absorption spectrometry). In the initial samples there were also determined (7) clay, silt and sand fractions (Robinson pipette method). Methods 1–3 and 6 and 7 are fully described by Van Reeuwijk [36], method 4 by Balbino [37] and method 5 by Jones [38].

Tissue samples (leaves, stems, fruit pulps) and samples of the organic amendments used in the experimental design were subjected to elemental analysis by Kjeldahl (N), colorimetry (B and P), and atomic absorption spectrophotometry (K, Ca, Mg, Fe, Mn, Cu, Zn, Ni, Cd, Cr, and Pb) methods [39] after tissue samples were digested with nitric acid in a microwave. In the samples of the organic amendments pH$_{H_2O}$ and conductivity were also determined [36].

2.5. Data Analysis

Data were firstly tested for normality and homogeneity of variances using the Shapiro-Wilk test and Bartlett's test, respectively. The comparison of the effect of the fertilizer treatments was provided by one-way ANOVA. When significant differences were found ($\alpha < 0.05$), the means were separated by the multiple range Tukey HSD test ($\alpha = 0.05$). The three depths at which the soil was sampled were treated as blocks in the analysis of variance of the variables related to soil properties.

3. Results

Accumulated olive yield was significantly higher in the High N in comparison to the other treatments, mainly due to the contributions of the olive yields of 2019 and 2020 (Figure 2). In 2017 and 2018 no significant differences were found between the fertilizer treatments. The organic amendments FYM and MSW provided the lower average total olive yields, although without significant differences for Control and Ash + N treatments.
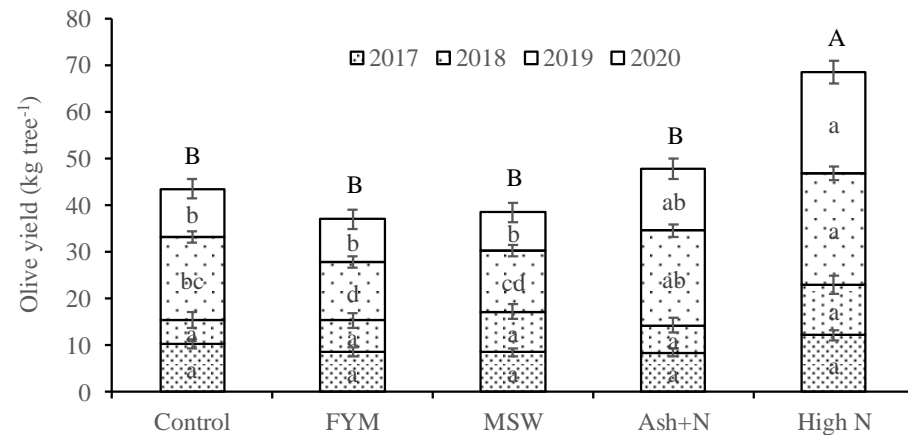

Figure 2. Olive yield in four consecutive harvests as a function of fertilizer treatments: control, compound NPK (50 kg ha^{-1} of N, P$_2$O$_5$ and K$_2$O); FYM, farmyard manure (rate equivalent of 50 kg N ha^{-1}); MSW, municipal solid waste (rate equivalent of 50 kg N ha^{-1}); Ash + N, bottom ash (4 t dw ha^{-1}) plus 50 kg N ha^{-1}; and High N, high N rate (120 kg N ha^{-1} and 50 kg ha^{-1} P$_2$O$_5$ and K$_2$O). Within each year (lowercase) and total (uppercase), means followed by the same letter are not significantly different by Tukey HSD test (α = 0.005). Vertical bars are standard errors.

Pruning wood displayed a pattern similar to that observed for the olive yield (Figure 3). In 2019 and 2020, the High N treatment showed significantly higher values than most of the other treatments, which resulted in total pruning wood significantly higher than in the FYM, MSW and Ash + N treatments. As observed for the olive yield, no significant differences between treatments were found in 2017 and 2018.
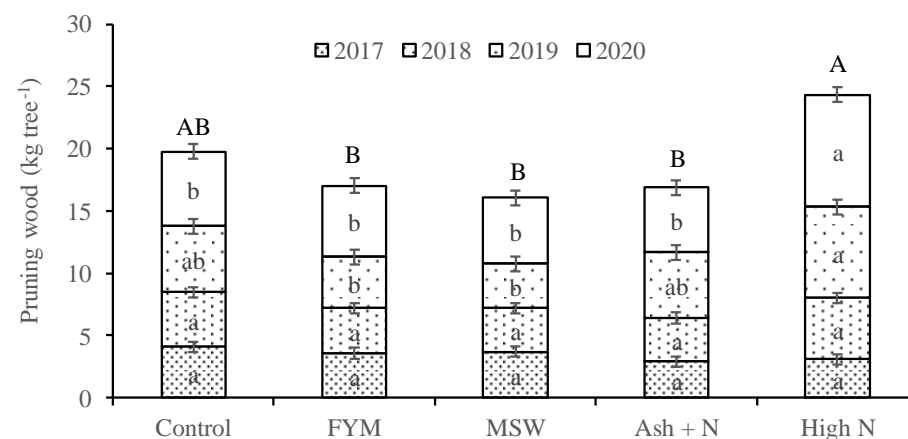

Figure 3. Pruning wood from four consecutive pruning events as a function of fertilizer treatments: control, compound NPK (50 kg ha^{-1} of N, P$_2$O$_5$ and K$_2$O); FYM, farmyard manure (rate equivalent of 50 kg N ha^{-1}); MSW, municipal solid waste (rate equivalent of 50 kg N ha^{-1}); Ash + N, bottom ash (4 t dw ha^{-1}) plus 50 kg N ha^{-1}; and High N, high N rate (120 kg N ha^{-1} and 50 kg ha^{-1} P$_2$O$_5$ and K$_2$O). Within each year (lowercase) and total (uppercase), means followed by the same letter are not significantly different by Tukey HSD test (α = 0.005). Vertical bars are standard errors.

The response of leaf gas exchange variables to the applied fertilizer treatments varied with the monitored dates (Figure 4). Regarding net photosynthetic rate, significant differences among treatments were only recorded on the third and fourth year of the study. High N trees presented the highest A in July of 2019 and 2020, but the trend was reversed in August and with higher evidence in September of 2020, in a strictly association with stomatal conductance values. In general, trees treated with organic soil amendments showed net photosynthetic rates similar to those fertilized with control NPK dose. Meanwhile, A/gs varied significantly between fertilizer treatments in four of the nine dates. In general, data highlighted the values of High N treatment, with tendency to higher A/gs in three dates, when leaves presented gs lower than 200 mmol m^{-2} s^{-1}, and the lower A/gs relatively to all organic amendment's treatments in July 2019 when gs of their leaves was higher than 200 mmol m^{-2} s^{-1}.
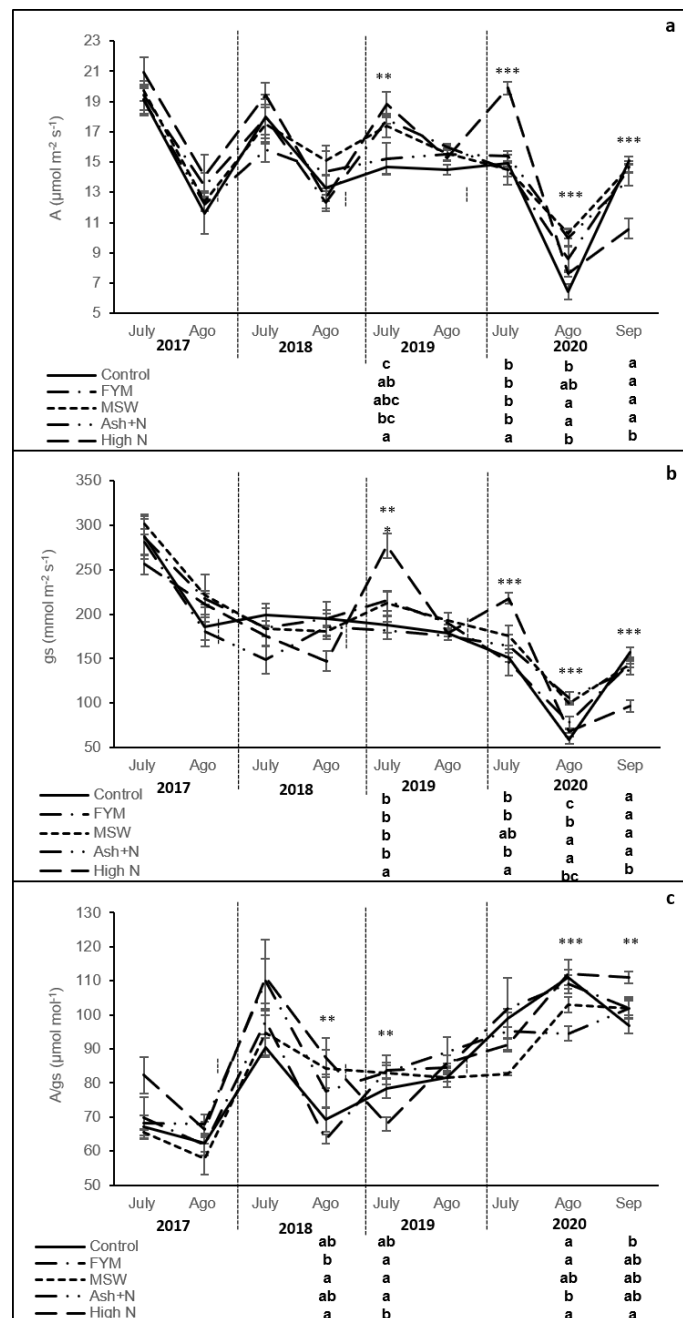

Figure 4. Net photosynthetic rate (**a**), stomatal conductance (**b**) and intrinsic water use efficiency (**c**) from July 2017 to September 2020 as a function of fertilizer treatments: control, compound NPK

(50 kg ha^{-1} of N, P$_2$O$_5$ and K$_2$O); FYM, farmyard manure (rate equivalent of 50 kg N ha^{-1}); MSW, municipal solid waste (rate equivalent of 50 kg N ha^{-1}); Ash + N, bottom ash (4 t dw ha^{-1}) plus 50 kg N ha^{-1}; and High N, high N rate (120 kg N ha^{-1} and 50 kg ha^{-1} P$_2$O$_5$ and K$_2$O). ** ($p < 0.01$) and *** ($p < 0.001$) are the results of analysis of variance. Within each date, means followed by the same letter are not significantly different by Tukey HSD test ($\alpha = 0.005$). Vertical bars are standard errors.

Leaf N concentration varied significantly between fertilizer treatments in five of the seven dates of samplings (Figure 5). The values of the High N treatment appeared systematically at the top of the figure, while the lines of FYM and MSW tended to be observed at the bottom of the figure. In general, the values appeared positioned in the lower middle part or even below the lower limit of the sufficiency range. Leaf P levels also differed significantly between treatments in five of the seven sampling dates. In this case, Ash + N, MSW and FYM appeared frequently at the top of the figure, whereas High N treatment frequently appeared at the bottom. Leaf P concentrations were generally found within the sufficiency range and only occasionally drop close to the lower limit. As for leaf N and P, leaf K concentrations varied significantly between treatments in five of the seven sampling dates. However, leaf K values showed greater variation between sampling dates and a more irregular pattern, but with a tendency to appear closer to the lower limit, in relation to the sufficiency range. When comparing treatments, the dominant pattern is the values of FYM at the top and the values of High N at the bottom of the figure. Leaf B levels differed significantly between treatments in two of the seven sampling dates. High N and Control treatments showed the higher values when significant differences between treatments were observed. In general, leaf B levels were found very close or below the lower limit of the sufficiency range. In general, no significant differences were found between treatments for other macro (Ca and Mg) and micronutrients (Fe, Zn, Cu and Mn), or these results revealed little consistency between sampling dates, having been considered of little relevance for this study (data not shown).
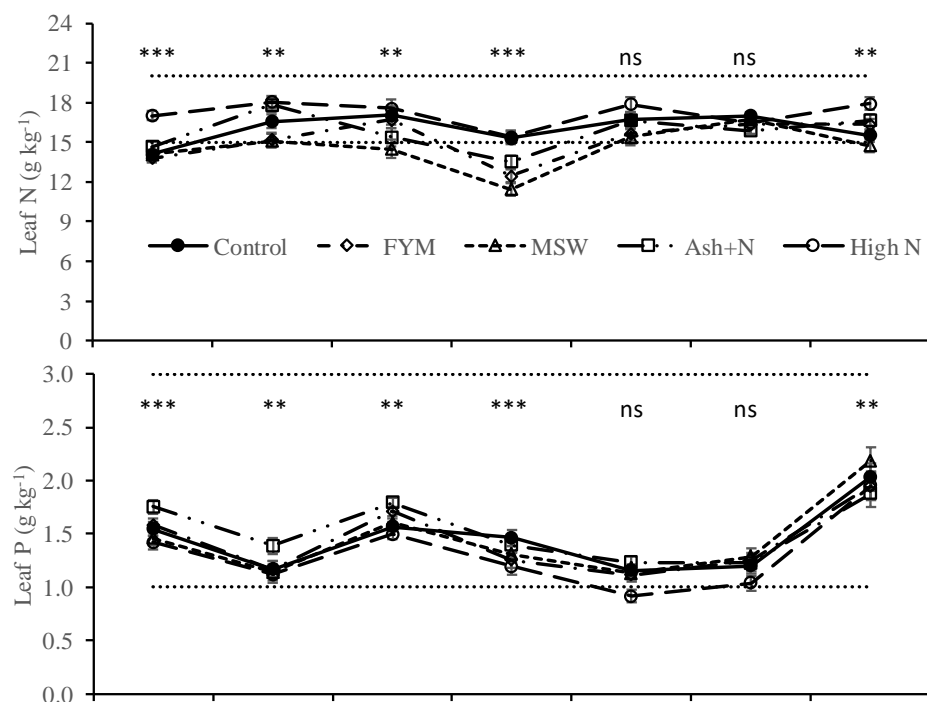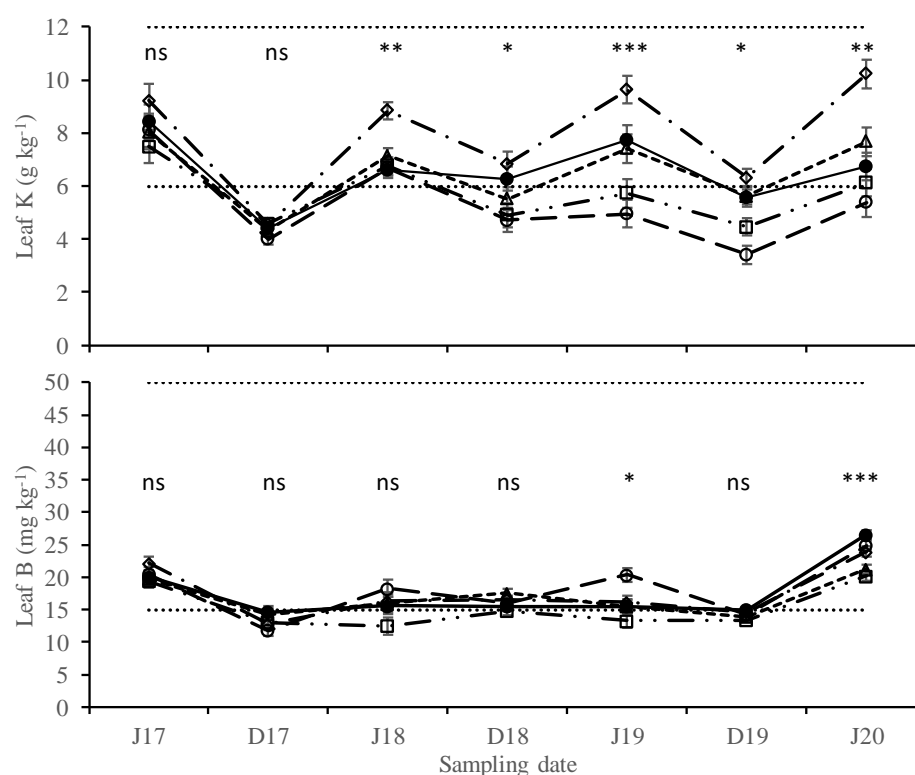

Figure 5. *Cont.*

Figure 5. Leaf nitrogen (N), phosphorus (P), potassium (K) and boron (B) concentrations in seven consecutive sampling dates in July (J) and December (D) from July 2017 (J17) to July 2020 (J20). Horizontal dashed lines are the lower and higher limits of the sufficiency ranges; ns (not significant), * ($p < 0.05$), ** ($p < 0.01$) and *** ($p < 0.001$) are the results of analysis of variance.

The concentration of Cd, Cr, Pb and Ni in the leaves varied little between treatments. However, a slight trend towards lower values in the High N and Control treatments was observed. The sampling date on which the differences between treatments were most accentuated was July 2018, following the second application of fertilizers and amendments (Table 3).

Table 3. Leaf concentration of trace metals in the sampling of July 2018, following the second application of fertilizers and amendments.

	Cadmium	Chromium	Lead	Nickel
Control	0.54 ab	2.88 b	5.15 a	9.03 ab
FYM	0.60 ab	3.83 ab	4.38 a	11.87 a
MSW	0.62 ab	4.29 a	4.92 a	11.51 a
Ash + N	0.81 a	4.07 a	6.09 a	12.62 a
High N	0.46 b	2.73 b	4.01 a	7.29 b

In columns, means followed by the same letter are not significantly different according to the Tukey HSD test ($\alpha = 0.05$).

The olive pulp was also analysed for elemental composition. Significant differences between treatments were uncommon and the ranges of variation were lower than those recorded on the leaves. Values of Pb and Cd in olive pulp were below 0.3 and 0.2 mg kg^{-1}, respectively, the threshold limits for edible vegetables as set by the Codex Alimentarius Commission [40]. The levels of Cr and Ni were also below to those usually found in several edible vegetables [41].

Relevant soil properties, such as organic C, pH, extractable P, K, B and Zn and cation exchange capacity significantly decreased from the surface to the deeper layers (Table 4). Organic C varied significantly between treatments. The High N treatment showed the

lowest values. Soil pH showed the trend of organic C, the lower values being found in the High N treatment, and the higher values in the MSW and FYM treatments. The higher values of extractable P and K were found in the MSW and FYM treatments and the lower values in the High N and Ash + N treatments, respectively. The treatments consisting of inorganic fertilizers (High N and Control) also showed reduced CEC, but increased soil B levels. Soil Zn levels were particularly high in the MSW treatment. Several other soil properties were determined but the results did not vary with the treatments and were considered of little relevance for this study (data not shown).

Table 4. Organic carbon (C), pH(H$_2$O), extractable phosphorus (P) and potassium (K), exchangeable calcium (Ca), magnesium (Mg) and K, cation exchange capacity (CEC) and extractable boron and zinc in soil samples taken in June 2020.

	Organic C (g kg^{-1})	pH$_{(H_2O)}$	Extrac. P (mg P$_2$O$_5$ kg^{-1})	Extrac. K (mg K$_2$O kg^{-1})	Exch Ca	Exch. Mg (cmol$_c$ kg^{-1})	Exch. K	CEC	Boron (mg kg^{-1})	Zinc
Soil depth (Z)										
0.0–0.1 m	11.6 a	6.2 a	185.6 a	282.6 a	4.9 a	0.9 a	0.6 a	7.9 a	1.3 a	4.1 a
0.1–0.2 m	8.3 b	6.0 b	88.7 b	152.1 b	3.9 b	0.9 a	0.3 b	6.8 ab	0.9 ab	2.0 ab
0.2–0.3 m	5.2 c	5.9 b	43.7 b	96.2 b	3.3 b	0.8 a	0.2 b	6.2 b	0.6 b	1.6 b
Treatment (T)										
Control	8.5 ab	5.8 bc	71.5 bc	142.7 bc	3.5 bc	0.7 c	0.3 b	5.6 b	1.7 a	1.4 b
FYM	9.2 ab	6.3 a	150.8 ab	350.7 a	3.9 bc	1.1 a	0.8 a	7.2 ab	0.4 b	2.0 b
MSW	10.1 a	6.4 a	216.1 a	170.6 bc	5.6 a	0.9 ab	0.3 b	8.8 a	0.3 b	6.4 a
Ash + N	7.8 bc	5.9 b	57.1 bc	73.8 c	4.2 b	0.9 ab	0.2 b	7.9 a	0.3 b	1.6 b
High N	6.2 c	5.6 c	34.3 c	147.1 bc	2.9 c	0.8 bc	0.3 b	5.5 b	1.9 a	1.4 b
Prob > F (Z)	<0.0001	<0.0001	<0.0001	<0.0001	<0.0001	0.0703	<0.0001	0.0055	0.0016	0.0182
Prob > F (T)	<0.0001	<0.0001	<0.0001	<0.0001	<0.0001	0.0001	<0.0001	<0.0001	<0.0001	0.0002

Within soil depth or treatment, means followed by the same letter are not significantly by Tukey HSD test (α = 0.05).

4. Discussion

The application of N at high rate (High N treatment) significantly increased olive yield and also had a strong influence on tree development as measured by pruning wood, particular in the last two years of the experiment. N concentration in the leaves, usually higher in the High N in comparison to the other treatments and the general positioning of the values close to the lower limit of the sufficiency range, showed N as the nutritional factor with greatest influence on the crop productivity. The experiment was installed in a Leptosol of low content of clay and organic matter, and, thus, reduced N holding capacity, since clays of type 2:1 and organic matter are the main mechanisms by which soils accumulate N that becomes gradually available to plants [4]. This makes these trees very dependent on the regular application of N as a fertilizer. Even though in some studies results have been reported in which no differences in olive yield were observed by the application of N [42,43]. Nonetheless, in poor fertility soils it has been shown that regular N application is decisive to maintain the growth and productivity of olive trees [44–46].

The treatments consisting of mineral fertilization (High N and Control) also received B, which appeared reflected in the levels of B in the soil and in the concentration of B in plant tissues. Considering that leaf B levels were generally low, close or below the lower limit of the sufficiency range, it is likely that B also has had some effect on the performance of the trees. The importance of B in dicot species is high [47] and in the experimental site region the application of B to olive trees has proved to be an important factor for productivity [44,48].

The effect of P, K and other nutrients in crop growth and yield seemed less relevant than that of N. In the case of P, some treatments, mainly MSW and FYM, increased its levels in the soil, but much less in plant tissues, perhaps because the trees tend to regulate the concentration of P in the leaves, by accumulating the nutrient in the roots [49,50]. The levels of P in the leaves were generally within the sufficiency range, which is in accordance with the extensive research on P fertilization in olive and other crops in the region where it has been difficult to obtain a response to the application of P [41,49,51]. Tissue K levels varied

greatly between sampling dates, which is a feature of this nutrient, especially because it is removed in high amounts in fruits [52,53], and due to its prominent role in the transport of photosynthates to growing tissues [11]. However, K leaf levels usually did not drop below 4 g kg^{-1}, the critical value for the olive tree's response to the application of K [53,54]. The remaining nutrients did not vary significantly between treatments and the values remained within the sufficiency ranges, so their effect on trees in this study seemed to be reduced.

The responses of crop yield and tree growth to the application of N at high rate (High N treatment) during the last two years of the experiment were associated with the higher photosynthetic activity of these trees in situations where stomatal conductance values overcome $150 \text{ mmol m}^{-2} \text{ s}^{-1}$, confirming the causal relationship between N nutrition and photosynthesis, as shown by other studies [55–57], including in olive trees [46,58]. The photosynthetic capacity is related to the nitrogen content primarily because the proteins of the Calvin cycle and thylakoids represent most of the leaf nitrogen [59]. Nonetheless, it is important to note that on the last two sampling dates, namely on the final one, after a period of particularly severe drought stress and sharp drop in gs, High-N trees showed the lowest net photosynthetic rates, indicating that higher N application increased plant susceptibility to water stress conditions, as found for other crops [60–62]. Thus, in view of altered precipitation patterns and reduced water availability due to climate change, careful adoption of nitrogen fertilization is required to ensure adequate productivity under rainfed conditions. Furthermore, overall, results of A/gs support the findings of other studies, presented in the review of Brueck [63], where N supply had positive or no effects on intrinsic water use efficiency, suggesting that non-stomatal or both stomatal and mesophylic limitations explain the N effects on A/gs.

Organic amendments (MSW and FYM) revealed a low contribution to the productivity performance of the trees, perhaps due to not having ensured an adequate supply of N to the plants. Organic amendments sometimes show low nutrient use efficiency, because instead of being mineralized, nitrogen can remain in organic form for long periods or the release of nutrients occurs when the opportunity for root uptake is low [3,64]. Organic amendments have increased the organic C content in the soil, which helps to support the previous statement. Through the application of organic amendments, the pH also increased, which may reflect more the initial high pH of the products, and less the effect of mineralization and nitrification, as their result tend to be an acidifying process [4]. Organic amendments also increased extractable P and CEC. However, although all these variables are positive aspects for soil fertility, in the short term they did not have a relevant influence on crop productivity. This does not rule out the possibility of benefits that could be obtained in the long-term as a result of their continued use.

The Ash + N treatment tended to show values that rarely stood out in comparison to the treatments of inorganic or organic fertilization, which seems in accordance with its initial composition. In general, bottom ashes are fertilizing materials that can be valued for their content in nutrients such as P, K, Ca or Mg [20–22], but which can also present toxicity problems as they may contain high levels of heavy metals, such as Cd, Cr or Pb [23–26]. In this study, neither aspect deserves to be highlighted, perhaps reflecting, once again, its initial mineral composition and the moderate rates in which it was applied.

A detail that deserves particular attention is the fact that the High N treatment has reduced the soil organic matter content. The increase of N rates stimulates the growth of herbaceous vegetation, which should be associated with an increase in the content of organic matter in the soil due to the increased deposition of fresh organic debris. However, soil tillage in Spring may have limited the opportunity for weeds growth, thus reducing the apparent advantage of the High N treatment. In general, soil tillage is frequently associated with reduced organic matter in the soil in comparison to other ground management systems that permit a better development of herbaceous vegetation [32,65]. However, this argument seems to be insufficient to clarify the situation since in the other inorganic fertilization treatments no reduction in soil organic C was found in comparison to the initial situation of the study four years before. In the High N treatment, an effect known as added N

interaction (formerly priming) appears to have occurred. Added N interaction reflects a stimulus of the inorganic N in soil biological activity, leading to an increased mineralization of the native organic matter of the soil [66,67]. The phenomenon was reported by Rodrigues and colleagues [14] when they found a reduction in soil organic C in the subsurface layers of a cover crop of annual legumes in comparison to a cover of natural vegetation. The result was attributed to the increased availability of inorganic N in the soil, resulting from the mineralization of the legume debris in the superficial layers, which accelerated the mineralization of the native soil organic matter. Thus, although these four years' results have been very positive for the High N treatment regarding crop growth and yield, soil organic matter turnover deserves attention in future studies to assess whether the use of inorganic N in high rates, mainly in soil management systems that do not allow the entry of organic debris into the soil, does not compromise the long-term sustainability of the production system.

In this study, heavy metals such as Cd, Cr, Pb or Ni were not an important concern. Although the MSW used in this study had some legal restrictions to be used in vegetable crops [19], due to the risk of containing some of those heavy metals at high level, and also bottom ash, a product sometimes associated with heavy metal contamination, as above mentioned, levels of heavy metals found in the soil did not differ between treatments and were within ranges acceptable for agricultural activity, as reported in other studies [41,68]. The values of heavy metals in the leaves rarely differed between treatments, and the values found in olive fruit pulp were within the safety standards for edible food [40].

5. Conclusions

Soil N availability probably was the most determining factor for the growth and yield of the olive trees, mainly because the experimental site soil had reduced N reserves, due to the low content of organic matter and also of clay, the latter being very important in the accumulation of inorganic N in the ammoniacal form. Thus, the High N treatment resulted in higher olive yields and the treatments consisting of organic amendments (MSW and FYM) were associated with poorer N nutritional status of olive trees. Organic amendments, however, increased the organic matter content in the soil, as well as P levels and CEC, which could play an important role in the long-term if this fertilization strategy is maintained over the years. Inorganic fertilization with a high N rate, associated with a soil management system that did not allow the development of herbaceous vegetation, significantly reduced the organic matter content of the soil due to a previously reported phenomenon known as added N interaction, which can compromise the long-term sustainability of this fertilization strategy. There was no increase in heavy metals in soil or plant tissues, associated with potentially more dangerous products such as MSW and bottom ash, so their use in olive groves can be recommended even though their effects should be continuously monitored.

References

1. Rusinamhodzi, L.; Dahlin, S.; Corbeels, M. Living within their means: Reallocation of farm resources can help smallholder farmers improve crop yield and soil fertility. *Agric. Ecosyst. Environ.* **2016**, *216*, 125–136. [CrossRef]
2. Bashagaluke, J.B.; Logah, V.; Opoku, A.; Sarkodie-Addo, J.; Quansah, C. Soil nutrient loss through erosion: Impact of different cropping systems and soil amendments in Ghana. *PLoS ONE* **2018**, *13*, e0208250. [CrossRef]
3. Havlin, J.L.; Tisdale, S.L.; Nelson, W.L.; Beaton, J.D. *Soil Fertility and Fertilizers, an Introduction to Nutrient Management*, 8th ed.; Pearson: Boston, MA, USA, 2014.
4. Weil, R.R.; Brady, N.C. *Nature and Properties of Soils*, 15th ed.; Pearson: London, UK, 2017.
5. Yang, X.; Zhang, P.; Li, W.; Hu, C.; Zhang, X.; He, P. Evaluation of four seagrass species as early warning indicators for nitrogen overloading: Implications for eutrophic evaluation and ecosystem management. *Sci. Total Environ.* **2018**, *635*, 1132–1143. [CrossRef] [PubMed]
6. Poikane, S.; Phillips, G.; Birk, S.; Free, G.; Kelly, M.G.; Willby, N.J. Deriving nutrient criteria to support 'good' ecological status in European lakes: An empirically based approach to linking ecology and management. *Sci. Total Environ.* **2019**, *650*, 2074–2084. [CrossRef] [PubMed]
7. Coyne, M.S. Biological denitrification. In *Nitrogen in Agricultural Systems*; Agronomy Monograph n.°49; Schepers, J.S., Raun, W.R., Eds.; ASA, CSSA, SSSA: Madison, WI, USA, 2008; pp. 201–253.
8. Pelster, D.E.; Larouche, F.; Rochette, P.; Chantigny, M.H.; Allaire, S.; Angers, D.A. Nitrogen fertilization but not soil tillage affects nitrous oxide emissions from a clay loam soil under a maize–soybean rotation. *Soil Tillage Res.* **2011**, *115/116*, 16–26. [CrossRef]
9. Li, G.; Huang, G.; Li, H.; van Ittersum, M.K.; Leffelaar, P.A.; Zhang, F. Identifying potential strategies in the key sectors of China's food chain to implement sustainable phosphorus management: A review. *Nutr. Cycl. Agroecosyst.* **2016**, *104*, 341–359. [CrossRef]
10. Gilbert, N. The disappearing nutrient. *Nature* **2009**, *461*, 716–718. [CrossRef] [PubMed]
11. Hawkesford, M.; Horst, W.; Kichey, T.; Lambers, H.; Schjoerring, J.; Moller, I.S.; White, P. Function of macronutrients. In *Marschner's Mineral Nutrition of Higher Plants*; Marschner, P., Ed.; Elsevier: London, UK, 2012; pp. 135–189.
12. Agegnehu, C.; Nelson, P.N.; Bird, M.I. Crop yield, nutrient uptake and soil physicochemical properties under organic soil amendments and nitrogen fertilization on Nitisols. *Soil Tillage Res.* **2016**, *160*, 1–13. [CrossRef]
13. Murtaza, B.; Zaman, G.; Imran, M.; Shah, G.M.; Amjad, M.; Ahmad, N.; Naeem, M.A.; Zakir, A.; Farooq, A.; Ahmad, S.; et al. Municipal solid waste compost improves crop productivity in saline-sodic soil: A multivariate analysis of soil chemical properties and yield response. *Commun. Soil Sci. Plant Anal.* **2019**, *50*, 1013–1029. [CrossRef]
14. Rodrigues, M.A.; Dimande, P.; Pereira, E.; Ferreira, I.Q.; Freitas, S.; Correia, C.M.; Moutinho-Pereira, J.; Arrobas, M. Early-maturing annual legumes: An option for cover cropping in rainfed olive orchards. *Nutr. Cycl. Agroecosyst.* **2015**, *103*, 153–166. [CrossRef]
15. Obriot, F.; Stauffer, M.; Goubard, Y.; Cheviron, N.; Peres, G.; Eden, M.; Revallier, A.; Vieublé-Gonod, L.; Houot, S. Multi-criteria indices to evaluate the effects of repeated organic amendment applications on soil and crop quality. *Agric. Ecosyst. Environ.* **2016**, *232*, 165–178. [CrossRef]
16. Pardo, R.; Schweitzer, J.P. *A Long-Term Strategy for a European Circular Economy—Setting the Course for Success*; Policy Paper Produced for the Think 2030 Project; Institute for European Environmental Policy: Brussels, Belgium, 2018.
17. Leogrande, R.; Lopedota, O.; Vitti, C.; Ventrella, D.; Montemurro, F. Saline water and municipal solid waste compost application on tomato crop: Effects on plant and soil. *J. Plant Nutr.* **2016**, *39*, 491–501. [CrossRef]
18. Rodrigues, M.A.; Garmus, T.; Arrobas, M.; Gonçalves, A.; Silva, E.; Rocha, L.; Pinto, L.; Brito, C.; Martins, S.; Vargas, T.; et al. Combined biochar and organic waste have little effect on chemical soil properties and plant growth. *Span. J. Soil Sci.* **2019**, *9*, 199–211. [CrossRef]
19. EU (European Union). Regulation (EU) 2019/1009, of the European Parliament and the Council of 5 June 2019. *Off. J. Eur. Union* **2019**, *170*, 1–114.
20. Brod, E.; Haraldsen, T.K.; Breland, T.A. Fertilization effects of organic waste resources and bottom wood ash: Results from a pot experiment. *Agric. Food Sci.* **2012**, *21*, 332–347. [CrossRef]
21. Schönegger, D.; Gómez-Brandón, M.; Mazzier, T.; Insam, H.; Hermanns, R.; Leijenhorst, E.; Bardelli, T.; Juárez, M.F.-D. Phosphorus fertilising potential of fly ash and effects on soil microbiota and crop. *Resour. Conserv. Recycl.* **2018**, *134*, 262–270. [CrossRef]
22. Martinez-Santos, T.; Bonfim-Silva, E.M.; Silva, T.J.A.; Damasceno, A.P.A.B. Correction of soil compaction using wood ash in safflower crop. *Aust. J. Crop. Sci.* **2019**, *13*, 1375–1382. [CrossRef]
23. Dahl, O.; Nurmesniemi, H.; Pöykiö, R.; Watkins, G. Heavy metal concentrations in bottom ash and fly ash fractions from a large-sized (246 MW) fluidized bed boiler with respect to their Finnish forest fertilizer limit values. *Fuel Process. Technol.* **2010**, *91*, 1634–1639. [CrossRef]
24. Jayaranjan, M.L.D.; van Hullebusch, E.D.; Annachhatre, A.P. Reuse options for coal fired power plant bottom ash and fly ash. *Rev. Environ. Sci. Biotechnol.* **2014**, *13*, 467–486. [CrossRef]
25. Maschowski, C.; Zangna, M.C.; Trouvé, G.; Gieré, R. Bottom ash of trees from Cameroon as fertilizer. *J. Appl. Geochem.* **2016**, *72*, 88–96. [CrossRef]
26. Ciesielczuk, T.; Dulewska, C.R.; Poluszyńska, J.; Ślęzak, E.; Łuczak, K. Ashes from sewage sludge and bottom sediments as a source of bioavailable phosphorus. *Ecol. Eng.* **2018**, *19*, 88–94. [CrossRef]

27. Bhattacharya, S.S.; Iftikar, W.; Sahariah, B.; Chattopadhyay, G.N. Vermicomposting converts fly ash to enrich soil fertility and sustain crop growth in red and lateritic soils. *Resour. Conserv. Recycl.* **2012**, *65*, 100–106. [CrossRef]

28. Merino, A.; Omil, B.; Fonturbel, M.T.; Vega, J.A.; Balboa, M.A. Reclamation of intensively managed soils in temperate regions by addition of wood bottom ash containing charcoal: SOM composition and microbial functional diversity. *Appl. Soil Ecol.* **2016**, *100*, 195–206. [CrossRef]

29. Fonseca, J.A.; Hanisch, A.L. Is biomass ash an effective product for use in cereal crops in an agroecological system? *Rev. Ciênc. Agrovet.* **2018**, *17*, 454–461. [CrossRef]

30. Romanowska-Duda, Z.; Janas, R.; Grzesik, M. Application of Phytotoxkit in the quick assessment of ashes suitability as fertilizers in sorghum crops. *Int. Agrophys.* **2019**, *33*, 145–152. [CrossRef]

31. Alcántara, C.; Soriano, M.A.; Saavedra, M.; Gómez, J.A. Sistemas de manejo del suelo. In *El Cultivo del Olivo*, 7th ed.; Barranco, D., Fernández-Escobar, R., Rallo, L., Eds.; Mundi-Prensa: Madrid, Spain, 2017; pp. 335–417.

32. Rodrigues, M.A.; Arrobas, M. Cover cropping for increasing fruit production and farming sustainability. In *Fruit Crops: Diagnosis and Management of Nutrient Constraints*; Srivastava, A.K., Hu, C., Eds.; Elsevier: Amsterdam, The Netherlands, 2020; pp. 279–295.

33. Fraga, H.; Atauri, I.G.C.; Malheiro, A.; Moutinho-Pereira, J.; Santos, J.A. Viticulture in Portugal: A review of recent trends and climate change projections. *OENO One* **2017**, *51*, 61–69. [CrossRef]

34. Yang, C.; Fraga, H.; van Ieperen, W.; Trindade, H.; Santos, J.A. Effects of climate change and adaptation options on winter wheat yield under rainfed Mediterranean conditions in southern Portugal. *Clim. Chang.* **2019**, *154*, 159–178. [CrossRef]

35. von Caemmerer, S.; Farquhar, G.D. Some relationships between the biochemistry of photosynthesis and the gas exchange of leaves. *Planta* **1981**, *153*, 376–387. [CrossRef] [PubMed]

36. Van Reeuwijk, L.P. *Procedures for Soil Analysis*; Technical Paper 9; ISRIC FAO: Wageningen, The Netherlands, 2002.

37. Balbino, L.R. *La Méthode Egner-Riehm et la Détermination du Phosfore et du Potassium «Assimilável» des Sols du Portugal. II Col. Medit. Cont. Fert. Plantas Cultivadas*; Facultad de Ciencias: Sevilla, Spain, 1968; pp. 55–65.

38. Jones, J.B., Jr. *Laboratory Guide for Conducting Soil Tests and Plant Analysis*; CRC Press: Boca Raton, FL, USA, 2001.

39. Temminghoff, E.E.J.M.; Houba, V.G. *Plant Analysis Procedures*; Kluwer Academic Publishers: Dordrecht, The Netherlands, 2004.

40. FAO/WHO (Codex Alimentarius Commission). Joint FAO/WHO Food Standards Programme, Codex Committee on Contaminants in Foods, Food 2018, CF/12 INF/1, 1–169. In Proceedings of the 12th Session of the Codex Committee on Contaminants in Foods, Utrecht, The Netherlands, 12–16 March 2018.

41. Arrobas, M.; Afonso, S.; Ferreira, I.Q.; Moutinho-Pereiram, J.M.; Correia, C.M.; Rodrigues, M.A. Liming and application of nitrogen, phosphorus, potassium and boron on a young plantation of Chestnut. *Turk. J. Agric. For.* **2017**, *41*, 441–451. [CrossRef]

42. Fernández-Escobar, R.; Marin, L.; Sánchez-Zamora, M.A.; García-Novelo, J.M.; Molina-Soria, C.; Parra, M. Long-term effects of N fertilization on cropping and growth of olive trees and on N accumulation in soil profile. *Eur. J. Agron.* **2009**, *31*, 223–232. [CrossRef]

43. Fernández-Escobar, R.; García-Novelo, J.M.; Molina-Soria, C.; Parra, M.A. An approach to nitrogen balance in olive orchards. *Sci. Hortic.* **2012**, *135*, 219–226. [CrossRef]

44. Rodrigues, M.A.; Pavão, F.; Lopes, J.I.; Gomes, V.; Arrobas, M.; Moutinho-Pereira, J.; Ruivo, S.; Cabanas, J.E.; Correia, C.M. Olive yields and tree nutritional status during a four year period without nitrogen and boron fertilization. *Commun. Soil Sci. Plant Anal.* **2011**, *42*, 803–814. [CrossRef]

45. Haberman, A.; Dag, A.; Shtern, N.; Zipori, I.; Erel, R.; Ben-Gal, A.; Yermiyahu, U. Significance of proper nitrogen fertilization for olive productivity in intensive cultivation. *Sci. Hortic.* **2019**, *246*, 710–717. [CrossRef]

46. Ferreira, I.Q.; Arrobas, M.; Moutinho-Pereira, J.M.; Correia, C.M.; Rodrigues, M.A. The effect of nitrogen applications on the growth of young olive trees and nitrogen use efficiency. *Turk. J. Agric. For.* **2020**, *44*, 278–289. [CrossRef]

47. Broadley, M.; Brown, P.; Cakmak, I.; Rengel, Z.; Zhao, F. Function of nutrients, micronutrients. In *Marschner's Mineral Nutrition of Higher Plants*; Marschner, P., Ed.; Elsevier: London, UK, 2012; pp. 191–248.

48. Ferreira, I.Q.; Rodrigues, M.A.; Arrobas, M. Soil and foliar applied boron in olive: Tree crop growth and yield, and boron remobilization within plant tissue. *Span. J. Agric. Res.* **2019**, *17*, e0901. [CrossRef]

49. Ferreira, I.Q.; Rodrigues, M.A.; Moutinho-Pereira, J.M.; Correia, C.; Arrobas, M. Olive tree response to applied phosphorus in field and pot experiments. *Sci. Hortic.* **2018**, *234*, 236–244. [CrossRef]

50. Rodrigues, M.A.; Piroli, L.B.; Forcelini, D.; Raimundo, S.; Domingues, L.C.; Cassol, L.C.; Correia, C.M.; Arrobas, M. Use of commercial mycorrhizal fungi in stress-free growing conditions of potted olive cuttings. *Sci. Hortic.* **2021**, *275*, 109712. [CrossRef]

51. Afonso, S.; Arrobas, M.; Ferreira, I.Q.; Rodrigues, M.A. Leaf nutrient concentration standards for lemon verbena (Aloysia citrodora Paláu) obtained from field and pot fertilization experiments. *J. Appl. Res. Med. Aromat. Plants* **2018**, *8*, 33–40. [CrossRef]

52. Rodrigues, M.A.; Ferreira, I.Q.; Claro, A.M.; Arrobas, M. Fertiliser recommendations for olive based upon nutrients removed in crop and pruning. *Sci. Hortic.* **2012**, *142*, 205–211. [CrossRef]

53. Fernández-Escobar, R. Fertilization. In *El Cultivo del Olivo*, 7th ed.; Barranco, D., Fernández-Escobar, R., Rallo, L., Eds.; Mundi-Prensa: Madrid, Spain, 2017; pp. 419–460. (In Spanish)

54. Ferreira, I.Q.; Arrobas, M.; Moutinho-Pereira, J.M.; Correia, C.; Rodrigues, M.A. Olive response to potassium applications under different water regimes and cultivars. *Nutr. Cycl. Agroecosyst.* **2018**, *112*, 387–401. [CrossRef]

55. Dwyer, L.M.; Anderson, A.M.; Stewart, D.W.; Ma, B.L.; Tollenaar, M. Changes in maize hybrid photosynthetic response to leaf nitrogen, from pre-anthesis to grain fill. *Agron. J.* **1995**, *87*, 1221–1225. [CrossRef]

56. Reddy, A.R.; Reddy, K.R.; Padjung, R.; Hodges, H.F. Nitrogen nutrition and photosynthesis in leaves of pima cotton. *J. Plant Nutr.* **1996**, *19*, 755–770. [CrossRef]

57. Correia, C.M.; Moutinho-Pereira, J.M.; Coutinho, J.F.; Björn, L.O.; Torres-Pereira, J.M.G. Ultraviolet-B radiation and nitrogen affect the photosynthesis of maize: A Mediterranean field study. *Eur. J. Agron.* **2005**, *22*, 337–347. [CrossRef]

58. Boussadia, O.; Steppe, K.; Zgallai, H.; Ben El Hadj, S.; Braham, M.; Lemeur, R.; Van Labeke, M.C. Effects of nitrogen deficiency on leaf photosynthesis, carbohydrate status and biomass production in two olive cultivars 'Meski' and 'Koroneiki'. *Sci. Hortic.* **2010**, *123*, 336–342. [CrossRef]

59. Evans, J.R. Photosynthesis and nitrogen relationships in leaves of C$_3$ plants. *Oecologia* **1989**, *78*, 9–19. [CrossRef] [PubMed]

60. Badr, M.A.; El-Tohamy, W.A.; Zaghloul, A.M. Yield and water use efficiency of potato grown under different irrigation andnitrogen levels in an arid region. *Agric. Water Manag.* **2012**, *110*, 9–15. [CrossRef]

61. Gheysari, M.; Loescher, H.W.; Sadeghi, S.H.; Mirlatifi, S.M.; Zareian, M.J.; Hoogenboom, G. Water-yield relations and water use efficiency of maize under nitrogen fertigation for semiarid environments: Experiment and synthesis. *Adv. Agron.* **2015**, *130*, 175–229.

62. Kiani, M.; Gheysari M- Mostafazadeh-Fard, B.; Majidi, M.M.; Karchani, K.; Hoogenboom, G. Effect of the interaction of water and nitrogen on sunflower under drip irrigation in an arid region. *Agric. Water Manag.* **2015**, *171*, 162–172. [CrossRef]

63. Brueck, H. Effects of nitrogen supply on water-use efficiency of higher plants. *J. Plant Nutr. Soil Sci.* **2008**, *171*, 210–219. [CrossRef]

64. Rodrigues, M.A.; Pereira, A.; Cabanas, J.E.; Dias, L.; Pires, J.; Arrobas, M. Crops use-efficiency of nitrogen from manures permitted in organic farming. *Eur. J. Agron.* **2006**, *25*, 328–335. [CrossRef]

65. Almagro, M.; de Vente, J.; Boix-Fayos, C.; García-Franco, N.; Aguilar, J.M.; González, D.; Solé-Benet, A.; Martínez-Mena, M. Sustainable land management practices as providers of several ecosystem services under rainfed Mediterranean agroecosystems. *Mitig. Adapt. Strateg. Glob. Chang.* **2016**, *21*, 1029–1043. [CrossRef]

66. Jenkinson, D.S.; Fox, R.H.; Rayner, J.H. Interactions between fertilizer nitrogen and soil nitrogen—The so-called "priming effect". *J. Soil Sci.* **1985**, *36*, 425–444. [CrossRef]

67. Schnier, H.F. Nitrogen-15 recovery fraction in flooded tropical rice as affected by added nitrogen interaction. *Eur. J. Agron.* **1994**, *3*, 161–167. [CrossRef]

68. Micó, C.; Peris, M.; Recatalá, L.; Sánchez, J. Baseline values for heavy metals in agricultural soils in an European Mediterranean region. *Sci. Total Environ.* **2007**, *378*, 13–17. [CrossRef] [PubMed]